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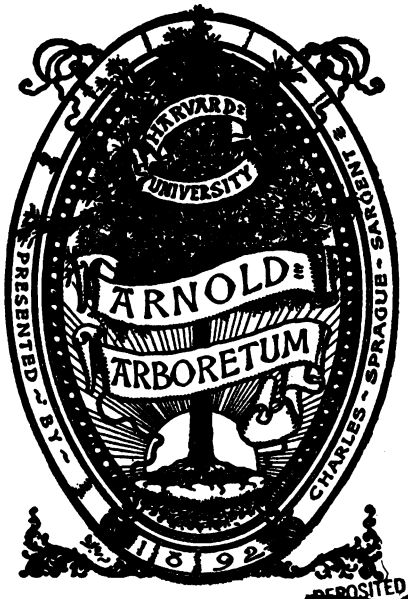

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R. E. DOOLITTLE, Acting Chief of Bureau.

PULP AND PAPER

AND OTHER PRODUCTS FROM WASTE RESINOUS WOODS.

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PULP AND PAPER AND OTHER PRODUCTS FROM WASTE RESINOUS WOODS.

INTRODUCTION.

The idea that the waste long-leaf yellow pine, Norway pine, Douglas fir, and other woods rich in resins can be used for the making of paper, wood turpentine, rosin oils, and similar products is not new, but the industry is just beginning to develop. It has received more or less attention in this bureau during the past seven years, where information as to its feasibility and practicability and as to the yields and character of the products which may be made has been gathered. This information points the way to the means whereby valuable timber may be conserved and the menacing waste wood of the lumbering operations may be profitably utilized.

The making of paper from these woods presents no unusual difficulties; in fact, several mills are running almost exclusively on such woods, while others use them in greater or less quantity. The recovery of wood turpentine, pine oils, and other products from resinous woods by steam or destructive distillation has been fairly well worked out, and, when a plant is properly located and equipped and operated skillfully, this industry is proving profitable.

The production of rosin oils, tars, and pitches from common rosin is also a thoroughly established, well-understood, and profitable industry.

With these successful results in view, it is at once clear that a combination of the three industries, using waste wood as the raw material from which all the products of these industries may be made, should be more profitable and require less capital, equipment, and labor than the three when conducted separately. Since each of these industries, paper-making, wood distillation, and rosin-oil production, is already well developed and the details quite well understood, no experimental demonstration on a manufacturing scale is needed to prove the practicability of the combination of the three. In short, such combination under one management is the most logical industrial condition.

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EXPERIMENTAL WORK.

MATERIAL AND PROCESS.

The experiments to be described were conducted with what is known in the South as "lightwood," which is long-leaf yellow pine that has lain in the forest until practically all the sap wood has decayed, leaving the heart wood sound. Such wood is rough, full of knots, and the greater part of it more or less charred from forest fires. For the experiments both the freshly prepared and the steam-extracted chips obtained from a steam wood turpentine plant were carefully selected to represent the average wood received at the plant.

The percentage of rosin which the chips contained was determined by extracting with ether and drying the extract in the water oven. The fresh chips contained 18.5 per cent of rosin on the basis of the water-free wood, while the chips which had been steamed at the turpentine plant contained 19.3 per cent. The total oil recovered from the black liquor was approximately 2 to 4 per cent lower than these figures.

The results are applicable only to average lightwood. Yields will naturally vary with the kind of wood employed. A cord of slabs and lap will yield less turpentine, rosin, or rosin oils than a cord of lightwood, while freshly split old stumps will yield more; old slabs and lap yield less than the fresh or green. In other words, the yields differ in quantity but not in kind with the character of the wood employed. The wood of long-leaf pine saw timber contains, as a rule, from 2 to 6 per cent of resin and about 0.2 to 0.5 per cent of turpentine and the short-leaf pine saw timber about the same. It would probably not be profitable to attempt to recover rosin oils and by-products from wood containing as little as 2 per cent of resin. The value of the by-products from such wood will rarely reach $3 per cord, and even in well-designed and efficient works it is doubtful if a profit can be secured from them.

The experimental cooking was done in a small rotary digester holding about 4 kilograms (8.8 pounds); from 2 to 4 kilograms of air-dry wood were cooked at a charge. Wood and dilute alkali were placed in the digester, which was then rotated to insure perfect contact and heated during rotations until the pressure reached 40 to 50 pounds, when the digester was connected with a condenser and water and turpentine distilled at the stated pressure for the time stated in Table 1. The digester was then closed and again rotated for from 15 to 20 minutes at 40 to 50 pounds pressure and again relieved as before, this procedure being repeated four or five times. The condensed water and turpentine were received in a graduated constant level vessel in which the turpentine could be accurately measured

after separating from the water. The volume of water condensed was also noted. After the turpentine had ceased to distill in measurable quantities the wood was cooked at different temperatures for various periods of time. The chips as received from the chipper were very irregular in size and shape, varying from coarse dust to pieces an inch thick and several inches long. They were screened into three sizes—those smaller than ¼ inch mesh, 31 per cent; those ¼ to 1 inch mesh, 44 per cent; and those larger than 1 inch mesh, 25 per cent of the wood. This separation was made in order to obtain data on the quantity of the various products that could be obtained from each of the several sizes and the time required to steam off the turpentine and to cook properly the chips of different sizes.

All the steaming data are found in Table 1. These include the amount of air-dry and moisture-free wood employed in each experiment; the volume of dilute alkali solution added; the volume of water and of turpentine and also the percentage of the total turpentine and the time and pressure each time the digester was relieved; the total volume of water and turpentine recovered in relieving and the actual time of relieving; the percentage of crude turpentine on the basis of the moisture-free wood; the yield of crude turpentine estimated to a cord of 4,000 pounds of air-dry wood assumed to contain 20 per cent of moisture; and the specific gravity and refractive index at 20° C. of the crude turpentine. These data are given on the screened fresh and steamed chips and also on the unscreened steamed chips.

TABLE 1.—*Data on steaming wood in alkali.*

	Fresh chips.			Steamed chips.							
	Under ¼ inch mesh.	¼ to 1 inch mesh.	Over 1 inch mesh.	Unsorted.	Unsorted.	Unsorted.	Under ¼ inch mesh.	¼ to ½ inch mesh.	Under ¼ inch mesh.	¼ to 1 inch mesh.	Over 1 inch mesh.
	Cook 7.	Cook 6.	Cook 8.	Cook 1.	Cook 2.	Cook 3.	Cook 4.	Cook 5.	Cook 9.	Cook 10.	Cook 11.
Air-dry wood (grams)	2,500	2,500	2,000	2,000	4,000	3,000	3,000	1,350	2,000	2,000	2,000
Bone-dry wood (grams)	2,265	2,217	1,831	1,538	3,077	2,310	2,739	1,236	1,733	1,838	1,818
Total volume of soda solution added (cc)	9,530	9,489	8,492	4,409	13,335	9,311	9,912	6,067	8,316	8,558	8,544
STEAMING DATA.											
First relief: Time of distillation at 46 pounds (minutes)	30	30	30	60	60	90	15	.....	21	25	30
Water distilled (cc)	700	800	880	.....	2,000	1,500	500	500	1,000	1,000	1,000
Crude turpentine distilled (cc)	52	61	29	8.5	19	17	9	7	5	11.7	7.5
Crude turpentine distilled (per cent of total oil removed)	86	82	71	.....	.....	.....	60	66.6	100	66.6	50
Second relief: Time of distillation at 46 pounds (minutes)	20	30	30	.....	.....	.....	17	.....	31	29	30
Water distilled (cc)	670	900	900	.....	.....	.....	500	500	1,000	1,000	900
Crude turpentine distilled (cc)	5	8.4	5	.....	.....	.....	3	2	0	3.3	2.5
Crude turpentine distilled (per cent of total oil removed)	8	11	12	.....	.....	.....	20	19	0	19	16.6
Third relief: Time of distillation at 46 pounds (minutes)	25	20	30	.....	.....	.....	12	.....	25	25	20
Water distilled (cc)	730	600	900	.....	.....	.....	500	500	1,000	1,000	1,000
Crude turpentine distilled (cc)	2	3	4	.....	.....	.....	1	1	0	1.7	1.6
Crude turpentine distilled (per cent of total oil removed)	3	4	10	.....	.....	.....	6.6	9.5	0	9.5	11.1
Fourth relief: Time of distillation at 46 pounds (minutes)	23	25	45	.....	.....	.....	15	5	25	25	25
Water distilled (cc)	1,000	700	920	.....	.....	.....	500	500	1,000	1,000	1,100
Crude turpentine distilled (cc)	2	2	1	.....	.....	.....	1	.5	0	.8	1.6
Crude turpentine distilled (per cent of total oil removed)	3	2	2	.....	.....	.....	6.6	4.7	0	4.7	11.1
Fifth relief: Time of distillation at 46 pounds (minutes)	18	15	15	.....	.....	.....	12	.....	.....	.....	20
Water distilled (cc)	900	500	400	.....	.....	.....	350	1,000	.....	.....	1,000
Crude turpentine distilled (cc)	0	0	2	.....	.....	.....	0	0	.....	.....	2
Crude turpentine distilled (per cent of total oil removed)	0	0	4	.....	.....	.....	0	0	.....	.....	12

Total ater distilled (cc)	4,000	4,000	4,000	.....	2,000	1,500	2,350	3,000	4,000	4,000	5,000
Total crude turpentine distilled (cc)	60	74	41	8.5	19	17	15	10.5	5	17.5	16
Total time for distillation (minutes)	118	120	135	60	60	90	91	90	102	104	125
Crude turpentine on basis of dry wood (per cent)	2.4	3	2	.5	.6	.7	.5	.8	.3	.9	.7
Calculated yield per cord (4,000 pounds) containing 20 per cent water (gallons)	10.2	12.8	8.6	2.13	2.4	2.8	2.1	3.3	1.1	3.7	3.2
Specific gravity of oil 20° C./20° C	.....	.891	.....	.....	.....	.....	.....	.....	.9095	.9095	.9095
Refractive index at 20° C	1.475	1.473	1.473	1.475	1.471	1.481	1.481	1.472	1.480	1.480	1.480

STEAMING DATA.

It will be seen that when fresh chips were used approximately 86 per cent of the total crude oils recovered were obtained in the first 30 minutes' relieving from the chips passing a one-fourth-inch mesh; 82 per cent from the chips passing between a one-fourth and a one-inch mesh; while but 71 per cent were recovered from the chips larger than one inch. The recovery in subsequent relievings falls off rapidly from the fine chips, more slowly from the medium chips, and still more slowly from the coarse chips. Not all the turpentine was removed from the coarse chips. The total quantity of crude oils recovered was least from the coarse and greatest from the medium chips. There is no doubt, however, that the quantity of crude oils in the chips was least in the fine and greatest in the coarse, since the oils volatilize readily from the fine chips on exposure and very slowly from the coarse chips. After cooking for pulp in Cook 8, in which case the pressure was maintained for one hour at 75 pounds, 6 cc of crude turpentine were recovered in 600 cc of water. From the pulp of Cook 3, cooked under the same conditions, 3 additional cc of turpentine were recovered in 800 cc of water.

The quantity of crude oils actually recovered from the chipped lightwood under very favorable laboratory conditions was at the rate of from 10 to 13.2 gallons per 4,000 pounds of wood containing 20 per cent of moisture, this percentage being an assumed quantity, approximately the average percentage of moisture in air-dry, seasoned wood. This yield is considerably below the yield generally claimed by steam wood turpentine producers, but agrees quite closely with the experimental work of the laboratory at steam plants, and also with the census reports. Attention is called to the fact, however, that some losses probably occurred in the transportation of the chips from Florida to Washington, even though they were carefully wrapped in paper and boxed to prevent such loss. The figures are certainly not too high for the particular chips employed.

Data of the same general nature were obtained in the distillation of the steamed chips. A smaller percentage of the total oils recovered was obtained in the first relieving, but this percentage was greatest from the fine chips and least from the coarse. The recovery decreased more rapidly from the fine and medium than from the coarse chips. These figures also indicate the quantity of crude oils left in 4,000 pounds of each size of chips steamed in stationary retorts. This quantity is from 1 to 2 gallons in fine, 3¼ to 3¾ gallons in medium, and practically 4 gallons in coarse chips. In other words, from 2 to 3.3 gallons of crude oils per cord remain in ordinary lightwood chips, steamed in the usual way in upright retorts.

It will be observed that there is some variation in the total time of relieving and in the volume of water distilled among these experi-

ments. Close observation of the accumulation of the oils indicates that these variations were not sufficient to cause a material difference in the amounts of oil recovered.

In the experiments with fresh chips in the first relieving or the first stage of the distillation it required approximately 14 parts of water to remove 1 part of oil from the fine chips, 13 parts to remove 1 part from the medium chips, and 30 parts to remove 1 part from the coarse chips. The proportion of oils to condensed water decreased very rapidly and in the fourth or next to the last stage it required approximately 500 parts of water to remove 1 part of turpentine from the fine chips, approximately 350 parts to remove 1 part from the medium, and approximately 900 parts to remove 1 part from the coarse chips. Considering the whole distillation (four relievings) 1 part of oils was removed from the fine chips by approximately 51 parts of water, from the medium by 47 parts, and from the coarse by 90 parts. From the steamed chips 55 to 200 parts of water on the fine, 90 on the medium, and 125 on the coarse were required to remove 1 part of turpentine in the first stage, and from 500 to 1,000 parts were required in the final stage by chips of all sizes. Of the total oils recovered, 1 part required from 133 to 310 parts of water, the greatest proportion being required by the coarse chips.

It will also be seen from Table 1 that from chips which would pass an inch screen more than 90 per cent of the recovered turpentine was removed in two relievings and that the total time of steaming to accomplish this was from 1 to 1¼ hours. In Cook 7, chips small enough to pass a one-fourth inch screen required 1,370 cc of water to remove 57 cc of turpentine from 2,500 grams of chips; that is, 94 per cent of the turpentine was recovered by an average of 1 pound of steam to 1.8 pounds of wood. In Cook 6, on chips passing an inch screen, but not the one-fourth inch screen, 1,700 cc of water removed 69 cc or 93 per cent of the recovered turpentine from 2,500 grams of chips, or an average of 1 pound of steam to 1.3 pounds of chips. In Cook 8, where the chips would not pass an inch screen, 1,780 cc of water removed 34 cc or 76 per cent, of the recovered turpentine from 2,000 grams of wood, or an average of 1 pound of steam to 1.1 parts of wood. It will also be observed (p. 12) that 6 cc or 8 per cent, of the recovered turpentine was recovered in Cook 8 in the subsequent pulp cook, where the digester was closed for 1 hour at a pressure of 75 pounds (160° C.). This turpentine was removed by 600 cc of water in 25 minutes' relieving.

In these experiments the purpose was to remove all wood turpentine and pine oil from the chips, and not to determine the best conditions for doing this. Steaming was continued until the proportion of turpentine to condensed steam or water was much smaller than is usual or would be profitable on a commercial scale. Apparently all light

oils, turpentine, and pine oils, which could be obtained under the conditions, were removed.

The data on the proportion of oils to water in the later relievings have, therefore, practical significance only as showing how far from being saturated with oils the steam was, and what working conditions should be avoided.

In the case of the fresh chips the proportions observed in the first relieving of the digester represent very well the proportions obtained on a commercial scale; that is, approximately 14 parts of water are used to remove 1 part of oils. Approximately this proportion has been observed at a well-managed steam wood turpentine plant. In this plant 1 pound of steam was used to 1.3 pounds of wood and approximately 12,000 pounds of water were required to remove 750 pounds of crude turpentine from the chips. It will be seen from a comparison of the percentage of oils steamed out of the fresh with that steamed out of the factory chips that this plant was recovering approximately 80 per cent of the crude turpentine in the chips. This statement is, of course, based on the assumption that the loss by volatilization of the carefully wrapped and boxed chips was no greater in transportation from Florida to Washington, D. C., than from storage between chipping and distilling. Whatever may have been the loss from this cause, and it is probably but a small fraction of 1 per cent, it is clear that approximately 0.5 per cent of oils was left in the wood at this plant, and that by far the larger part of these oils was readily recovered under the conditions of these experiments with the distillation of 100 to 200 parts of water to 1 part of oil; that is, there were left in a cord of wood 2 to 3 gallons of oils the removal of which required, under favorable experimental conditions, steam equal to from 200 to 500 gallons of water.

It has been observed by the Bureau of Chemistry in refining crude wood turpentine with saturated steam that the lighter oils, those that may properly be termed "wood turpentine," distill at the rate of approximately 1 part of turpentine to 1 part of water, but this proportion slowly changes throughout the distillation, and toward the close, and when the heavy oils or pine oils only are distilling, there is only 1 part of turpentine to 5 or more parts of water. For the distillation of practically the whole of any volume of ordinary crude wood turpentine the distillation of from three to four times as much water is necessary. At the best 10 to 15 parts of water to 1 part of turpentine were required in these experiments, and the same proportions have been observed at steam turpentine works.

In the distillation of crude turpentine from wood with steam it is desirable for economic working to approach these proportions as closely as possible, though it is almost certain that they can not be equaled and absolutely certain that they can not be materially

increased. Every effort should be made to have the steam carry as large a proportion of turpentine as is practicable. It is evident that if the proportion obtaining in refining can be had in steaming from the wood, steam consumption can be greatly reduced and the cost of equipping the plant and of making turpentine considerably lowered. Experiments are being conducted with this object in view. The experience and observations of the Bureau of Chemistry indicate that this condition can best be secured by relieving periodically from the retort. After the removal of the easily freed oils, which are very largely obtained in the first half hour of steaming without pressure, the valve in the outlet pipe to the condenser may be closed and the chips subjected to 40 to 50 pounds of steam pressure for from 20 to 30 minutes, when the outlet valve may be opened for an equal period, maintaining the pressure all the time. This steaming and relieving may be alternated until it is no longer profitable to steam the chips.

PULP-COOKING DATA.

An additional quantity of water was placed in the digester with the chips and the alkali solution to distill off all the turpentine. Thus, during the early stages of the cooking, while the turpentine was being steamed off, the cooking of the fiber was conducted at low pressure with a dilute alkali solution. The concentration of the alkali solution increased gradually through the distillation of water and turpentine, until for the final stage the cooking was conducted in a 20 to 27 per cent alkali solution. During the steaming period the pressure never exceeded 50 pounds, while the digester was closed and did not fall below 25 to 35 pounds during relieving.

All of the cooking data so far as they relate directly to the production of pulp have been brought together in Table 2.

TABLE 2.—*Cooking data.*

Cook No.	Size of chips.	Cooked below 50 pounds.	Cooking pressure.		Alkali.				
			Pressure.	Time.	Strength final cook.	Causticity.	Grams per liter.	Consumed.	Excess.
		Hours.	*Pounds.*	*Hours.*	*Per cent.*	*Per ct.*		*Per ct.*	*Per ct.*
1	Unsorted	2.25	100	2.5	27.3	97.7	81	15.3	11.8
2	do	.....	100	1.5	27.0	98.2	80	.....	.....
3	do	1.75	75	1.0	27.0	98.3	100	11.8	15.2
4	Less than ¼ inch	1.66	75	1.0	20.0	98.0	69	12.9	7.1
5	¼ to ½ inch	2.50	75	1.0	20.0	98.0	80	.....	.....
6	½ to 1 inch	2.75	75	1.0	20.0	98.0	80	11.3	8.7
7	Less than ¼ inch	2.75	75	1.0	20.0	97.6	80	14.7	5.3
8	Larger than 1 inch	2.75	75	1.0	20.0	98.0	80	10.1	9.9
9	Less than ¼ inch	2.66	75	1.0	20.0	98.0	80	12.8	7.2
10	¼ to 1 inch	2.66	75	1.0	20.0	98.1	80	12.3	7.7
11	Larger than 1 inch	2.75	75	1.0	20.0	98.2	80	10.2	9.8

TABLE 2.—*Cooking data*—Continued.

Cook No.	Size of chips.	Dissolved by sodium hydroxid.	Yield dry unbleached fiber.	Remarks on fiber.
		Per ct.	*Per ct.*	
1	Unsorted			Well cooked, except large pieces, which were undercooked.
2	do			Small material overcooked; large material undercooked.
3	do			Large somewhat undercooked; stock long.
4	Less than ¼ inch			Overcooked.
5	¼ to ½ inch	57.0	43.0	Reduced well; long, tough fiber.
6	½ to 1 inch	55.0	45.0	Fair strength and length.
7	Less than ¼ inch	54.2	45.8	
8	Larger than 1 inch	43.2	56.8	Large chips not cooked.
9	Less than ¼ inch	56.0	44.0	Much fine overcooked stuff; large sufficiently cooked for wrapping paper.
10	¼ to 1 inch	52.8	47.2	Well cooked.
11	Larger than 1 inch	43.8	56.2	Large chips not cooked.

The wood as received, approximately 30 per cent of which would pass a one-fourth-inch screen and 25 per cent which would not pass an inch screen, could of course not be cooked to produce a uniform pulp. The fine material was overcooked and weak, while the large pieces were cooked only superficially, often remaining hard in the centers. These facts are shown by the results of cooks 1 to 3.

The cooking data show a low consumption of alkali and a high yield of fiber. This is due partly to the fact that the cook was made for the production of a strong wrapping paper, for which the wood employed was suitable only, because of the charcoal which is almost invariably present in lightwood. The pulp after milling was very firm and strong and the fiber long and well separated, in every way suitable for making strong wrapping papers. The average air-dry unbleached pulp approximated 45 to 50 per cent from wood which contained about 18 per cent of rosin. The total time of cooking was somewhat under the usual time required for cooking wood by the soda process. Probably the time required in the mill to steam off turpentine and to cook for a strong wrapping paper is slightly longer than that required in these small-scale experiments. This time will naturally differ considerably with mill conditions. In no case do the results indicate more than a normal loss of fiber through the action of the alkali. It is true that the yields of fiber from the finest material were lower than those from larger chips, though not differing greatly from the yields from chips larger than one-fourth and smaller than one inch. As previously stated, however, the pulp from the finer chips was quite weak and overcooked.

It has been established in paper mills and on a commercial scale that a very good grade of wrapping paper is made from long-leaf pine. Samples have been tested in the Bureau of Chemistry which show a strength factor of from 0.7 to 1. This is practically equal to similar paper made by chemical processes from spruce.

TREATMENT OF THE BLACK LIQUOR.

The black liquor and washings from the cooks were evaporated at atmospheric pressure, dried in vacuum, and weighed. The residue was then destructively distilled in round, hard-glass flasks, the products obtained being given in Table 3 on the basis of the dry liquor and also of the wood.

TABLE 3.—*Data on the dry distillation of black liquors.*

Products from distillation.	Fresh chips.			Steamed chips.		
	Cook 7.	Cook 6.	Cook 8.	Cook 9.	Cook 10.	Cook 11.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Dry liquor, on basis of dry wood (contains soda salts)	70.9	70.3	59.6	71.4	68.7	61.8
Oil distilled, on basis of dry liquor	23.3	23.9	24.2	23.4	23.7	20.4
Pyroligneous acid, on basis of dry liquor	17.2	18.4	12.5	14.1	14.2	14.2
Oil distilled, on basis of dry wood	16.5	16.8	14.4	16.7	16.3	12.6
Pyroligneous acid, on basis of dry wood	12.2	12.9	7.5	10.1	9.8	8.8

DISTILLATION OF OILS OBTAINED FROM THE DRY BLACK LIQUOR.

The oils produced in distilling the dry black liquor were shaken several times with a 9 per cent caustic soda solution until no more was dissolved, for the purpose of separating creosote and other alkali soluble oils, the alkali solution containing these being subsequently acidified to insure separation of the oils. The oils insoluble in alkali were then fractionally distilled from an ordinary still; that distilling below 250° C. was classed as rosin spirit, as is the commercial practice, and that distilling from 250° to 400° C., after the more or less complete separation of a crystalline body, retene, was classed as rosin oil. Retene was present in all the fractions distilling at from 355° to 400° C., the largest quantity distilling between 388° and 393° C., or at the boiling point of retene, 390° C. The retene was separated as far as practicable from the rosin oils by chilling and cold pressing, but it is relatively certain that this procedure did not completely separate it from the oils, which in consequence contained some of it dissolved in them.

The results of the separation of the crude oils by the stated methods are given in Table 4, and these are also stated as percentages on the basis of both the dried liquor and the moisture-free wood.

TABLE 4.—*Distillation data showing approximate composition of oils.*

Products separated from crude oils.	Fresh chips.			Steamed chips.		
	Cook 7.	Cook 6.	Cook 8.	Cook 9.	Cook 10.	Cook 11.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Phenoloids, on basis of total oils from dry liquor	23.1	16.9	18.2	21.6	18.0	22.4
Rosin spirit (to 250° C.), on basis of total oils from dry liquor	12.3	15.6	15.9	12.6	13.0	11.0
Retene, on basis of total oils from dry liquor	3.1	8.1	4.5	6.3	7.7	2.7
Rosin oil, on basis of total oils from dry liquor	61.5	59.4	61.4	5.95	61.2	63.9
Phenoloids, on basis of dry wood	3.8	2.8	2.6	3.1	2.9	2.8
Rosin spirit (to 250° C.), on basis of dry wood	2.0	2.6	2.3	2.2	2.1	1.4
Retene, on basis of dry wood	.52	1.4	.65	1.0	1.2	.3
Rosin oil, on basis of dry wood	10.1	10.0	8.9	9.8	10.0	8.1

The quantities of the several constituents separated from the crude oils which were obtained by distilling the black liquor show some rather decided variations when calculated on the basis of the dry liquor. Thus the phenoloid bodies vary from 16.9 to 23.1 per cent, light oils from 11 to 15.9 per cent, retene from 2.7 to 11.2 per cent, and rosin oils from 58.8 to 63.9 per cent. The cause of these variations is not definitely known, but it is believed that it lies in variations in the speed of distillation, variations in atmospheric pressure on different days, and in unavoidable errors of measurement, errors accentuated by the comparatively small volumes of products obtained.

From these data the approximate percentage of each of the constituents of the crude oil obtained from this particular lot of wood may be thus stated:

	Per cent.
Phenoloids	18
Light or rosin spirits	14
Retene	5
Heavy or rosin oils containing an unknown quantity of retene	61

There is no question as to the profitable utilization of at least two of these—the light oils or rosin spirits and the heavy or rosin oils. Both of these products find a ready market, the former as a paint and varnish vehicle and the latter in the preparation of printers' inks and greases of various kinds. For the latter purpose it would appear unnecessary to separate the retene, as it is quite unctuous and would assist in giving body to the grease. Wood creosote may be separated from the phenoloids or alkali soluble oils which also serve in the preparation of antiseptic solutions or washes.

When the several constituents are figured on the basis of the dry wood, it is found that by this procedure outlined the wood yielded approximately the following results:

	Per cent.
Phenoloids	3
Light oils (below 250° C.)	2
Heavy oils (above 250° C.)	10
Retene	0.6

CONSTITUENTS OF CRUDE PYROLIGNEOUS ACID.

In Table 5 are given the results showing the composition of the aqueous portion or the crude pyroligneous acid produced by dry distilling the black liquor. This aqueous portion of the distillate retains in solution and suspension, tar, tar acids, phenols, acetates, acetone, etc. Only (1) total free acid, calculated as acetic, (2) esters, (3) acetone, and (4) methyl alcohol were determined in the aqueous distillate.

The crude acid is first distilled by heating to 140° C. with 10 cc phosphoric acid, adding small portions of water and distilling until all the acid is over. This distillate is made to volume and used for the following determinations:

Free acetic acid.—Titrate 100 cc with standard sodium hydroxid, using phenolphthalein as indicator. Boil for the end point on account of any carbon dioxid present.

Esters.—Saponify 100 cc with excess of normal sodium hydroxid with reflux condenser for two hours, and titrate back with normal acid, using phenolphthalein as indicator. Run a blank with water. Subtract the number of cubic centimeters previously found to be required to neutralize the free acid, from the number of cubic centimeters of soda neutralized by the solution. The remainder is neutralized by the esters present. One molecule of sodium hydroxid (40 grams) equals one molecule of methyl acetate (74 grams).

Acetone.—Messinger's method:[1] Place 10 cc of the sample in a 500 cc glass stoppered bottle. Add 25 cc of normal sodium hydroxid and run in drop by drop 20 cc fifth-normal iodin. Let stand 15 minutes; add 26 cc normal sulphuric acid. The iodin in excess of the amount required to form iodoform (CHI_3) is then titrated with tenth-normal sodium thiosulphate. Three molecules of iodin (761) equal one molecule of acetone.

Methyl alcohol.—Zeisel method, as modified by Perkins and applied to the analysis of pyroligneous acid by Striter and Zeidler:[2] Distill 10 cc of the sample with 30 cc hydriodic acid. Pass the methyl iodid evolved through a train, the first flask of which contains 25 cc of 2 per cent potassium arsenite to free the methyl iodid from iodin and hydriodic acid. Each of the second and third flasks contains 20 cc alcoholic silver nitrate. The apparatus is filled with carbon dioxid and the glycerin bath containing the distilling flask heated up to about 125° to 127° C., or as high as possible without causing the hydriodic acid to distill. All of the methyl iodid should be over in 1 hour. Wash the contents of the silver nitrate bottles into a beaker, make to 450 cc, acidify with nitric acid, heat one-half hour and filter into a Gooch. Correct for the amount of methyl

[1] Klar. Technologie des Holzverkohlung, 1910, p. 364. [2] Analyst, 1904, 29: 318.

acetate present. One molecule of silver iodid (234) equals one molecule of methyl alcohol (32).

TABLE 5.—*Composition of pyroligneous acid.*

Products of distillation.	Fresh chips.			Steamed chips.		
	Cook 7.	Cook 6.	Cook 8.	Cook 9.	Cook 10.	Cook 11.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Total acids (calculated as acetic)	0.09	0.09	0.17	0.14	0.18	0.15
Methyl acetate	.19	.18	.16	.26	.19	.18
Methyl alcohol	3.74	4.64	6.60	5.06	5.80	4.97
Acetone	.95	.90	1.10	1.24	.93	1.03
Water	95.00	94.20	92.00	93.30	92.90	93.70
Total acids (as acetic) on basis of dry wood	.012	.013	.013	.014	.016	.015
Methyl acetate, basis of dry wood	.023	.025	.012	.026	.019	.016
Methyl alcohol, basis of dry wood	.468	.617	.505	.51	.51	.44
Acetone, basis of dry wood	.119	.120	.081	.125	.092	.09
Water, basis of dry wood	10.70	12.13	6.90	9.40	9.10	8.20

The concentration of methyl acetate, methyl alcohol, and acetone in the crude pyroligneous acid is much like that in the crude acid from hardwood distillation, and is sufficiently great to make the economical recovery of crude methyl alcohol feasible. The quantity of acids in the crude acid is too small, however, to be economically recovered. No comparison can be made between these yields of acid and the yields obtained from resinous woods by the ordinary methods of distillation. In these experiments the acetic acid formed from the wood combined immediately with the alkali, and in the subsequent distillation in the presence of an excess of soda was probably destroyed. Though the concentration of the methyl alcohol and acetone is high, the percentage yields from the wood are low, agreeing very well with the quantities usually obtained from resinous woods.

OTHER EXPERIMENTS WITH THE BLACK LIQUOR.

The black liquor was treated in several other ways. It was saturated at room temperature with carbon dioxid; then, under a pressure of 19 pounds, heated to 100° C., cooled and filtered. Filtration was slow when the mass became cold. The filtrate from the carbon dioxid precipitate gave a precipitate with hydrochloric acid. In a black liquor which contained 11.1 per cent of organic matter in solution, carbon dioxid precipitated 4.9 per cent and acetic acid precipitated 1.2 per cent from the filtrate. The carbon dioxid precipitate was dried and dry distilled. It yielded approximately 51 per cent of distillate, 17 per cent of water, 12 per cent of heavy oils, 11 per cent of light oils, and 6 per cent of creosote oils. The acetic acid precipitate did not contain all of the rosin removed from the wood by the alkali.

EXAMINATION OF THE CRUDE WOOD TURPENTINE.

In addition to the work previously described, the crude wood turpentine and light, heavy, and alkali-soluble oils were all roughly fractionated to obtain information concerning the general nature of these products.

The crude turpentine from cooks 6, 7, and 8 (fresh chips), and also that from cooks 9, 10, and 11 (steamed chips), were mixed to give two samples for distillation. The results obtained are given in Table 6.

TABLE 6.—*Fractionation of crude turpentine.*

Temperature.	Cooks 6, 7, and 8 (fresh chips).		Cooks 9, 10, and 11 (steamed chips).	
	Distillate.	Refractive index, 20° C.	Distillate.	Refractive index, 20° C.
°C.	*Per cent.*		*Per cent.*	
160	0	0	4	1.4660
160 to 170	[1] 21	1.466	16	1.4675
170 to 175	21	1.468	15	1.4680
175 to 180	11	1.469	7	1.4700
180 to 185	7	1.470	7	1.4710
185 to 190	4	1.472	..	.....
190 to 195	3	1.473	..	.....
195 to 200	3	1.474	..	.....
Residue.	30	1.488	51	.....

[1] Above 165° C.

RESULTS OF THE DISTILLATION OF ROSIN AND OF ROSIN AND SODA.

It was observed that the distillate from the dry distillation of the black liquor was lighter than that from the distillation of rosin alone. The general nature of the substance obtained from the redistillation of the products of the dry distillation of rosin and of those from the distillation of rosin in the presence of an excess of caustic soda is shown in the following table:

TABLE 7 —*Comparative data on the distillation of rosin and of rosin and soda.*

Fraction.	Total oil distilled.	Material.	Boiling points.	Specific gravity.	Refractive index.	Color.	Fluorescence.
	Per ct.		*°C.*				
1	10	Rosin.....	95 to 250	0.8750	1.475	Pale yellow.....	
1	21	Rosin and soda..	70 to 250	.8463	1.474	Very pale yellow..	
2	27	Rosin.....	250 to 335	.9842	1.536	Green.....	Slight blue.
2	44	Rosin and soda..	250 to 335	.9718	1.550	.....do.....	Very strong blue.
3	26	Rosin.....	335 to 350	1.0030	1.547	Yellowish green...	Blue.
3	14	Rosin and soda..	335 to 350	.9972	1.570	Greenish yellow...	Blue green.
4	24	Rosin.....	350 to 365	1.0240	1.552	Amber.....	Slight bluish green.
4	9	Rosin and soda..	350 to 365	1.0090	1.582	.....do.....	Green.
5	8	Rosin.....	365 to 375	.....	1.565	Red.....	Strong blue green.
5	6	Rosin and soda..	365 to 385	.....	1.595	.....do.....	Very strong green.

The volume of oils distilling below 335° C. is greater from mixed rosin and soda than from the rosin alone, and the specific gravity of these oils is lower than that of the oils produced from rosin alone, which distill below 335°. In other words, a greater quantity of lighter oils is obtained by distilling rosin with soda than by distilling rosin alone, 65 per cent of the total oils obtained distilling below 335° C. from the rosin and soda, while but 37 per cent distill from the rosin alone. It will be observed also that while a smaller percentage of oil distilling above 335° C. is obtained from the rosin and soda, the specific gravity of these oils is lower than that of the oils from rosin alone. The last three fractions from the rosin alone are thick and heavy, and flow with difficulty. All the fractions from the rosin and soda are limpid and free flowing. Results of the same nature were obtained from cooking the wood with soda. The results of the distillation of the black liquor are shown in Table 8.

FRACTIONATION OF THE OILS FROM THE BLACK LIQUOR.

The heavier oils (those heavier than water and distilling above 335° C.) obtained from the distillation of the black liquor were distilled from an ordinary side-neck distilling flask, thus roughly fractionating them, with the following results:

TABLE 8.—*Data obtained on fractionating the heavy oils distilled from the black liquor.*

Temperature.	Oil distilled.	Refractive index at 20° C.	Color.	Consistency.	Fluorescence.
°C.	*Per ct.*				
338 to 350	1.5	1.558	Yellow	Liquid	Slight.
350 to 361	8	1.585	.....	do	
361 to 372	20	1.596	Amber	Thick oil	Blue.
372 to 382	25	1.607	do	Paste	Do.
382 to 393	21	1.621	Reddish	Solid	Do.
393 to 404	8.5	1.628	Red	do	Do.
404 to 415	3	1.630	Dark red	do	Green.
415 to 426	3	1.635	do	do	Do.
426 to 439	2.2	1.640	do	Very thick oil	Do.
439 to 440	5	.....	do	do	Do.
440	1.5	.....	do	do	Do.

From 5 to 17 per cent of the heavy oil is retene, which may be partly separated by chilling and cold pressing. It was found that the distillation of the dried black liquor could be duplicated quite closely. Thus distillation from the same dried liquor gave 42, 41, and 43 per cent distillate.

The light oils obtained on distilling the black liquors were roughly fractionated from a side-tube flask in order to obtain some idea of their nature. The mixed oils from cooks 6, 7, and 8 gave the following results.

TABLE 9.—*Fractionation of light oils obtained in distilling the black liquors.*

Temperature.	Distillation.	Refractive index 20°C.	Temperature.	Distillation.	Refractive index 20°C.
°C.	*Per cent.*		*°C.*	*Per cent.*	
65 to 80	1	1.396	177 to 191	12	1.485
80 to 109	1	1.433	191 to 196	7	1.491
109 to 119	1	1.448	196 to 206	6	1.495
119 to 129	4	1.456	206 to 215	5	1.498
129 to 138	7	1.463	215 to 225	4	1.503
138 to 148	6	1.468	225 to 234	3	1.508
148 to 158	11	1.473	234 to 244	3	1.512
158 to 168	8	1.480	244 to 253	2	1.520
168 to 177	9	1.485	Residue.	9	1.551

OILS SOLUBLE IN 9 PER CENT CAUSTIC SODA SOLUTION.

As has been stated, the crude oils obtained from the black liquor were first washed repeatedly with 9 per cent caustic soda to remove phenoloid bodies and other oils soluble in soda. These were then precipitated from the soda solution with acid, washed with water and distilled.

Temperature (°*C*):	Distillate (per cent).
Up to 207	3
207 to 210	2
210 to 215	3
215 to 220	6

Distillation was continued up to 347°.

Another portion of these oils was distilled until the temperature reached 240° C. and this distillate roughly refractionated to obtain some idea of the percentage of guiacol and creosol in it. The following results were obtained:

Temperature (°*C.*):	Distillate (per cent).
Up to 207	5
207 to 210	5
210 to 215	8
215 to 220	9

The results indicate about 5 per cent of guiacol and about 27 per cent of guiacol and creosols.

PRODUCTS OBTAINED FROM LIGHTWOOD.

In Table 10 is given the percentage yield of all the recovered products on the basis of the moisture-free wood, both from the fresh and from the steamed chips.

These figures give an idea of the approximate quantities of the several constituents or products which may be recovered from ordinary lightwood when it is steamed to remove crude wood turpentine, cooked with soda to produce pulp, and the black liquor dried and distilled to produce oils and crude wood alcohol.

TABLE 10.—*Summary of yields of products from long-leaf yellow pine. (Results expressed on basis of moisture-free wood.)*

Products.	Fresh chips.			Steamed chips.							
	Cook 7.	Cook 6.	Cook 8.	Cook 1.	Cook 2.	Cook 3.	Cook 4.	Cook 5.	Cook 9.	Cook 10.	Cook 11.
	Below ¼ inch.	¼ to 1 inch.	Above 1 inch.	Unsorted.	Unsorted.	Unsorted.	Below ¼ inch.	¼ to ½ inch.	Below ¼ inch.	¼ to 1 inch.	Above 1 inch.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Crude wood turpentine	2.40	3.00	2.00	0.50	0.60	0.80	0.50	0.80	0.30	0.90	0.70
Turpentine distilling to 175°	.93	1.29	.98	.18	.20	.28	.17	.27	.11	.36	.31
Pine oils	1.48	1.78	1.35	.....	.....	.....	.....	.....	.15	.51	.44
Rosin spirit (to 250° C)	2.00	2.60	2.30	.....	.....	.....	.....	.....	2.20	2.10	1.40
Phenoloids	3.80	2.80	2.60	.....	.....	.....	.....	.....	3.20	2.90	2.80
Rosin oil	10.10	10.00	8.90	.....	.....	.....	.....	.....	9.80	10.00	8.20
Retene	.50	1.40	.70	.....	.....	.....	.....	.....	1.00	1.20	.40
Acetic acid	.01	.01	.01	.....	.....	.....	.....	.....	.01	.02	.02
Methyl acetate	.02	.03	.01	.....	.....	.....	.....	.....	.03	.02	.02
Methyl alcohol	.47	.62	.51	.....	.....	.....	.....	.....	.51	.51	.44
Acetone	.12	.12	.08	.....	.....	.....	.....	.....	.13	.09	.09
Water	10.70	12.10	6.90	.....	.....	.....	.....	.....	9.40	9.10	8.20
Fiber	45.80	45.00	56.80	.....	.....	.....	.....	43.30	43.90	47.20	56.20

SUPPLY OF WASTE RESINOUS WOODS.

The supply of waste resinous wood suitable for the manufacture of paper, turpentine, rosin, rosin oils, methyl alcohol, etc., can be only approximately estimated. The census figures for the lumber cut in 1910 are: Long-leaf pine, approximately 14,000,000,000 board feet; Douglas fir, 5,000,000,000 board feet; western pine, over 1,000,000,000 board feet; or a total of approximately 20,000,000,000 board feet. Authorities agree that at least 60 per cent of the tree as it stands in the forest is wasted in converting it into lumber, and that 25 per cent of the trees remain in the forests to rot or be destroyed in forest fires. That is, approximately 5,000,000 cords of waste wood are left annually in the forests in the lumbering of resinous woods, leaving out of consideration the dead and fallen timber in the uncut forest. This waste has been going on for many years. The sap or nonresinous part of the wood rots away in a few years, leaving the heart or resinous portion which will last indefinitely. Probably half of this annual forest waste becomes "lightwood," such as is used in the production of wood turpentine and tar. This material has been accumulating for years, and will probably continue to be added to for many more years.

In addition to this waste there is also a large source of supply in the stumps of cut-over lands and in the slabs and edging usually wasted at the mills. Altogether there are fully 8,000,000 cords of waste resinous woods annually produced in the lumber industry.

This waste wood would yield all the wrapping, building, and other low-grade colored papers, and all the rosin, rosin oils, turpentines, rosin spirits, and methyl alcohol which are now being produced in this country.

YIELDS FROM RESINOUS WOODS.

The following table shows the quantities of the several products which may be made from lightwood and from ordinary mill waste, both by cooking with caustic alkali, or first extracting with a volatile solvent and then cooking with alkali.

TABLE 11.—*Yields of various products from 1 cord (4,000 pounds) of long-leaf pine.*

Products.	From light wood.				From lean wood, mill waste, etc.	
	Alkali extraction.		Volatile solvents.		Alkali extraction.	Volatile solvents.
	Distills between—	Yield.	Distills between—	Yield.		
	°C.	*Gallons.*	*°C.*	*Gallons.*	*Gallons.*	*Gallons.*
Crude oils	154–400	36–120	154–250	8–20	20–50	2–10
Refined turpentine	150–180	5–20	150–180	6–16	2–8	1½–8
Pine oils	175–250	2–5	175–250	2–4	½–2	½–2
Rosin spirits	80–250	5–15			3–8	
Rosin oils	250–400	20–60			10–25	
Creosote	100–400	8–20			4–8	
Rosin				[1] 300–800		[1] 75–400
Methyl alcohol		½–4			½–5	
Calcium acetate		[1] 5–15			[1] 5–15	
Charcoal		[2] 10–25			[2] 10–25	
Paper pulp, unbleached		[1] 1,000–1,600		[1] 1,000–1,600	[1] 1,000–1,600	[1] 1,000–1,600

[1] Pounds. [2] Bushels.

The data that have come to the attention of the Bureau of Chemistry indicate that the products from Douglas fir and from Norway pine stumps fall within the limits given above for long-leaf pine. As a rule, seasoned stumps should approximate the higher yields from lightwood.

COST OF WASTE RESINOUS WOODS.

The cost of waste wood delivered at the mills in the South varies widely, but rarely exceeds $5 a cord. The Bureau of Chemistry has found that the average cost of lightwood delivered at the turpentine plants approximates $3.50 a cord. In case the wood is gathered by a lumbering company from its own forests and over its own tramroads this cost frequently does not exceed $2.50 and may fall as low as $1 a cord.

The stumps of long-leaf Norway pine and of Douglas fir, after the timber has been cut several years, are usually much richer in

resins than average lightwood. They are therefore especially suitable for the production of wood turpentine, rosin, and rosin oils. If care is taken to free them from earth, they are suitable for making paper. The cost of stump wood is often decidedly higher, especially in the West, than that of lightwood, because of the difficulty of removing stumps from the ground. This is best done by blasting, which has been found to cost approximately 5 cents a stump for longleaf pine of an average diameter of 13.6 inches.[1] Approximately 45 such stumps, 2½ feet tall, will yield a cord of wood, which makes the cost on the land approximately $2.25 a cord, which should be added to the cost of lightwood delivered at the mill, to give the approximate cost of stumps at the mill. The average cost of removing Douglas fir stumps[2] varying from 1 to 4 feet in diameter is about 84 cents each in Washington State, and 9 such stumps, averaging 3 feet in length and 2 feet in diameter, will yield a cord of wood. This makes the cost of the wood piled on the land ready to ship approximately $8 a cord, or possibly $10 a cord at the mill.

In estimating the cost of removing and gathering stumps the increased value of the land for agricultural purposes must be considered. The value of the land will in many instances, especially in the South, be increased sufficiently to pay for the cost of removing the stumps, and in other cases this increased valuation will greatly reduce the cost of the wood at the mill.

Paper makers will undoubtedly question the practicability of using stumps for making paper because of the earth adhering to them. This can be largely if not entirely removed by proper methods; and as material worth even less than wood a ton is profitably purified it appears reasonable to suggest that stump wood can also be profitably cleaned for paper making, at least in those regions far removed from the paper-making centers.

CONCLUSIONS.

It has been fully demonstrated, both in the laboratory and in the mill, that paper of good quality can be made from pine wood. The Bureau of Chemistry has called attention to this fact and to the industrial opportunities in this field in two former publications.[3]

It is feasible to combine three well-developed chemical industries—paper making, wood distillation (in a modified form), and the manufacture of rosin oils—and thus to obtain from a single raw material, waste resinous wood, practically all the valuable constituents which it contains. The country's sources of paper, turpentine, rosin oils,

[1] Mississippi Agr. Exp. Sta. Bul. No. 118.

[2] U. S. Dept. Agr., Bureau of Plant Industry Cir. 25.

[3] Circular 41. Papermaking Materials and their Conservation; Bulletin 144. Wood Turpentine: Its Production, Refining, Properties, and Use.

and wood alcohol can be greatly augmented and the injury to forests by fire and insects materially reduced by the utilization of this wood. It is believed that the results presented here will hold, approximately, for good average lightwood, except as to refined wood turpentine, which should run higher than here found. Especially is this true of the long-leaf lightwood of North Carolina, which, experience has shown, yields more wood turpentine than does the lightwood of Florida. This fact is possibly due in part to climate conditions, the longer and hotter summer of the South volatilizing more of the light oils.

The approximate yield for 4,000 pounds of cord air-dry wood (3,200 pounds moisture-free wood) of the valuable products and the value of each, together with the total value produced from a cord, is shown in the following table. The values are approximate wholesale values at the plant:

Refined wood turpentine, 6 gallons, at $0.40	$2.40
Pine oils, 7 gallons, at $0.35	2.45
Rosin spirits, 11 gallons, at $0.20	2.20
Rosin oils, 40 gallons, at $0.35	14.00
Phenoloids, 12 gallons, at $0.06	.72
Crude methyl alcohol, 3.5 gallons, at $0.35	1.20
Unbleached pulp, 1,440 pounds, at $0.0175	25.20
Total	48.17

Thus products worth $48.17 are made from wood which costs from $2 to $4 delivered at the works.

All these products are of good quality. The wood turpentine, pine oils, and rosin spirits are suitable paint and varnish thinners, especially for outdoor work; the rosin oils are suitable for making greases; the phenoloids are used for shingle strains and preservatives; and the pulp for making a good strong brown wrapping paper, quite similar to that now selling from 3 to 4 cents per pound.

As has been previously stated, there are works which are being profitably operated, making wood turpentine and pine oils alone from this wood. There are works profitably manufacturing rosin oils and rosin spirits from rosin made from the living trees, and others profitably making paper alone from wood. The combination of the three units into one industrial development appears, therefore, to be well worth the careful consideration of paper makers, lumbermen, investors and especially of those interested in the conservation of material resources through the utilization of waste. It is believed that such a combination offers the most profitable use of refuse wood and stumps on the cut-over pine lands of the South and West.

NOTE.—The bureau can not undertake to furnish details of equipment, nor estimates of cost of erecting and equipping mills for making paper and by-products from wood. The cost of erection varies greatly with local conditions, and those interested

should consult mill engineers for estimates and details of equipment and erection. These engineers advertise in the technical journals, especially in the paper-trade journals, and their names may be found in the directories of many of the larger cities.

It may be said, however, that estimates for the erection and equipping of paper mills, exclusive of water-power rights and wood rights, vary from $10,000 to $25,000 per day cord. From $500 to $2,000 should be added to these figures to cover the additional equipment required for the utilization of by-products, if the wood is cooked at once with caustic soda, and from $1,000 to $3,000 should be added if the resins are first extracted with a volatile solvent and turpentine and rosin made instead of turpentine and rosin oils. It is the opinion in this bureau that costs of erecting and equipping approximate the lower rather than the higher figures.

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